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STRENGTHENING MECHANISMS IN HIGH TEMPERATURE CERAMICS.(U)
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The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

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**20. ABSTRACT (Continue on reverse side if necessary and identify by block number)**

Research on Strengthening Mechanisms in High Temperature Ceramics was conducted in the Department of Metallurgy & Materials Science from 1 Sep 69 - 22 Feb 77. 16 Papers have appeared in the technical literature, and 4 more are presently being written; Research on 2 topics begun under these grants are continuing with support by other agencies. A list of papers published are included in the Appendix. ~~We briefly summarize~~ SUMMARIES OF the research accomplishments ARE LISTED AS FOLLOWS; ~~for convenience, they are separated into three areas~~--$Al_2O_3$, $MgAl_2O_4$ and $TiO_2$-$SnO_2$. Discussion of each is keyed to the Appendix.

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"Strengthening Mechanisms in High Temperature Ceramics"

by

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Research on Strengthening Mechanisms in High Temperature Ceramics was conducted in the Department of Metallurgy & Materials Science from September 1, 1969 through February 22, 1977 under sponsorship from the Army Research Office. During the course of this research, 16 papers have appeared in the technical literature, and a further 4 are presently being written; research on two topics begun under this grant are continuing with support by other agencies. A list of all papers published to date are included in the Appendix.

Five graduate students were supported for all or part of their graduate studies: Dr. B. J. Pletka (M.S. and Ph.D.), Dr. M. W. Park, (Ph.D.), and Mr. L. W. Hwang (M.S.). Messrs. W. D. Donlon and D. S. Phillips have began their research under ARO sponsorship but are still in graduate school.

In the following, we briefly summarize the research accomplishments; for convenience, they are separated into three areas--$Al_2O_3$, $MgAl_2O_4$ and $TiO_2$-$SnO_2$. Discussion of each is keyed to the Appendix, which lists the publications.

## 1. Plastic Deformation of Aluminum Oxide

As shown in the Appendix, ten publications have appeared in the open literature, and a further four are in preparation; work is continuing in this area by D. S. Phillips. Our first concerns were with hardening mechanisms in sapphire deforming by basal slip, which is the most easily activated slip system in alumina. Paper 1 presents a qualitative study of the dislocation substructure during basal slip, and was followed by a quantitative study, (papers 2 and 3), which led to a detailed model of work hardening in sapphire (paper 4). This theory also led to a model for creep in sapphire (paper 18). Concurrently, work was being done on deformation of Cr-doped and Ti-doped samples (papers 2, 5, and 17), which led to a general theory of solid solution hardening in ionic crystals (paper 6), where it was shown that aliovalent impurities are much more potent hardeners than isovalent impurities; the main contribution of the hardening comes from the elastic (rather than the electrostatic) interactions introduced by aliovalent impurities and their charge-compensating defects.

Ti-doped sapphire is also potentially a system in which precipitation hardening can be realized, since suitable heat treatment can lead to the formation of precipitates of oriented needles (star sapphire). Initial results of precipitation hardening are presented in paper 2 while the precipitation process itself is discussed in papers 7 and 8. The precipitate has now been definitively identified as rutile ($TiO_2$) (paper 19). Further studies on precipitation hardening in suitably-aged samples is underway in our laboratories and will also constitute part of D. Phillips Ph.D. work.

During the basal slip study, a unique climb dissociation of basal dislocations was observed (rather than the more familiar glide dissociation); this is described in paper 9, and a study of the energetics of the dissociation is described in paper 20.

In addition to the work on Ti-doped sapphire, work is continuing in our laboratory on work hardening of sapphire undergoing prismatic slip and the kinetics of dislocation dipole annhilation, both of which will be included in D. Phillips's Ph.D. thesis. Finally, the several slip systems in sapphire are discussed from a crystallographic point of view and compared to slip in MgO and $MgAl_2O_4$ in paper 10.

2. Plastic Deformation of Mg aluminate Spinel

Our studies of plastic deformation in spinel have been concerned mainly with understanding the impressive high temperatures strength of stoichiometric spinel, as discussed in paper 11, and the influence of non-stoichiometry on plastic deformation in spinel (paper 12). This work is continuing and will constitute the thesis work of Mr. Donlon; particular attention will be devoted to the role of climb in the high temperature deformation.

3. Spinodal Decomposition in $(Ti,Sn)O_2$

A study of decomposition in $(Ti,Sn)O_2$ solid solutions was undertaken to determine whether spinodal strengthening might be a useful strengthening mechanism for ceramics and papers 13-16 resulted from this effort. Although the technical aspects of preparing polycrystalline "alloys" that could be spinodally decomposed was sufficiently arduous that no actual study of spinodal strengthening was attempted, this work is believed to constitute the most detailed study of spinodal decomposition in a crystalline ceramic yet reported.

Paper 13 constitutes a redetermination of subsolidus equilibrium in this system and was necessitated by a controversy about the phase equilibria, which occurred during the initial stages of our research. Paper 14 is a theoretical analysis of small angle x-ray scattering. This tool has been widely used for studies of spinodal decomposition, and in this paper, we

clarify the interpretation to be given small angle x-ray spectra in spinodally decomposing systems. Papers 15 and 16 present the results of the studies of spinodal decomposition themselves, the former paper being concerned with the early stages of decomposition, and with alloys both inside and outside the spinodal, while the latter paper is concerned with coarsening of an alloy inside the spinodal.

In closing, the principle investigators want to acknowledge the unfettered support given by the Army Research Office over a considerable period of time, which allowed a most productive research program to be carried out.

Appendix: Publications resulting from AROD sponsored research


1. B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Dislocation Structures in Sapphire Deformed by Basal Slip," J. Am. Ceram. Soc. 57, 388 (1974).

2. B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Strengthening Mechanisms in Sapphire" in "Deformation of Ceramic Materials" (Plenum Press) p. 257 (1975).

3. B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Work Hardening and Creep Deformation of Corundum Single Crystals" in "Electron Microscopy in Mineralogy" (Springer-Verlag) p. 404 (1976).

4. B. J. Pletka, A. H. Heuer and T. E. Mitchell, "Work Hardening in Sapphire," Acta Met. 25, 25 (1977).

5. B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Solid Solution Hardening of Sapphire ($\alpha$-$Al_2O_3$)," Phys. Stat. Sol. (a) 39, 301 (1977).

6. T. E. Mitchell and A. H. Heuer, "Solution Hardening by Aliovalent Cations in Ionic Crystals," Mater. Sci. Eng. 27 (1977).

7. B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Precipitation in Titanium-doped Sapphire," Proc. Int. Conf. on Mechanical Behavior of Materials (Society of Materials Science, Japan) Vol IV, p. 413 (1972).

8. D. S. Phillips, B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Precipitation in Star Sapphire," in "Developments in Electron Microscopy and Analysis" (Academic Press) p. 493 (1976).

9. T. E. Mitchell, D. S. Phillips, B. J. Pletka and A. H. Heuer, "Climb Dissociation of Dislocations in Sapphire," Phl. Mag. 34, 441 (1976).

10. T. E. Mitchell, B. J. Pletka, D. S. Phillips, W. T. Donlon and A. H. Heuer, "Mechanical Properties of Oxide Single Crystals" in "Ceramic Microstructures 1976" (University of California Press).

11. L. Hwang, A. H. Heuer and T. E. Mitchell, "Slip in Stoichiometric $MgAl_2O_4$ Spinel" in "Deformation of Ceramic Materials" (Plenum Press) p. 257 (1975).

12. T. E. Mitchell, L. Hwang and A. H. Heuer, "Deformation in Spinel", J. Mater. Sci. 11, 264 (1976).

13. M. Park, T. E. Mitchell and A. H. Heuer, "Subsolidus Equilibria in the $TiO_2$-$SnO_2$ System," J. Am. Ceram. Soc. 58, 43 (1975).

14. M. W. Park, A. R. Cooper and A. H. Heuer, "Small Angle X-ray Scattering and the Linear Theory of Spinodal Decomposition," Scripta Met. 9, 321 (1975).

15. M. Park, T. E. Mitchell and A. H. Heuer, "Spinodal Decomposition in $TiO_2$-$SnO_2$," J. Mater. Sci. 11, 1227 (1976).

16. M. Park, T. E. Mitchell and A. H. Heuer, "Coarsening in a Spinodally Decomposing System: $TiO_2$-$SuO_2$" in "Electron Microscopy in Mineralogy" (Springer-Verlag) p. 205 (1976).

17. B. J. Pletka, T. E. Mitchell and A. H. Heuer, "Dislocation Structures in Deformed Sapphire Doped with Cr and Ti," in preparation.

18. B. J. Pletka, A. H. Heuer and T. E. Mitchell, "Creep of Sapphire," in preparation.

19. D. S. Phillips, T. E. Mitchell and A. H. Heuer, "The Nature of Precipitates in Star Sapphire," in preparation.

20. D. S. Phillips, B. J. Pletka, A. H. Heuer and T. E. Mitchell, "Energetics of Dipole Annealing in Sapphire," in preparation.